National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 928

Lead Nitrate

This Standard Reference Material (SRM) is certified for use as an assay standard for lead and is intended primarily for use in the calibration and standardization of procedures employed in analysis and for routine critical evaluation of the daily working standards used in these procedures. A unit of SRM 928 consists of 30 g of lead nitrate.

Certified Mass Fraction

Lead Nitrate [$Pb(NO_3)_2$]: 100.00 % ± 0.03 %

Certified Values: The certified value shown is based on the determination of lead in the material as received; drying being unnecessary. Lead is precipitated as the chromate using a slight excess of potassium dichromate (SRM 136c). The lead chromate is removed by filtration and the excess chromate ion determined spectrophotometrically. Details of this method are reported elsewhere [1]. The molecular weight of lead nitrate employed in the calculation is 331.219. This value is based on a mass-spectrometrically determined value of 207.209 for the atomic weight of lead in this sample. The uncertainty shown represents two standard deviations of a single measurement based on 16 determinations with allowances for known sources of possible error.

A semi-quantitative survey for trace metals by emission spectroscopy indicated the following: silver, 2 μg/g; chromium, 3 μg/g; and nickel 3 μg/g. No other metals were detected.

Expiration of Certification: The certification of **SRM 928** is valid, within the measurement uncertainty specified, for five years from the date of shipment from NIST, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Metrological Traceability: The measurand is the total mass fraction for lead nitrate. Metrological traceability is the SI derived unit for mass fraction (expressed as a percent).

Coordination of the technical measurements leading to the certification of SRM 928 was provided by I.L. Barnes formerly of NIST Inorganic Analytical Research Division.

Chemical analyses were performed by T.J. Murphy and J.W. Gramlich formerly of NIST of NIST Inorganic Analytical Research Division. Spectroscopic analyses were performed by C.S. Amell and D. Golightly of the U.S. Geological Survey.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

This Certificate of Analysis has undergone editorial revision to reflect program and organizational changes at NIST and at the Department of Commerce. No attempt was made to reevaluate the certificate values or any technical data presented on this certificate.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 23 February 2016
Certificate Revision History on Last Page

Steven J. Choquette, Acting Director
Office of Reference Materials

NOTICE AND WARNINGS TO USERS

This is intended for research use.

Storage: This SRM should be stored in the tightly-closed original bottle under normal laboratory conditions. Tests show the material to be dry as-received and will not adsorb appreciable water when exposed to a 90 % relative humidity atmosphere for 5 days.

INSTRUCTIONS FOR USE

Drying Instructions: No additional drying is required.

Preparation and Stability of Prepared Stock Solutions: The solutions of SRM 928 are stable as described below. At the time of use, these solutions should be clear and display no turbidity. Because of the instability of non-acidified aqueous lead solutions at the working levels it is recommended that three levels of concentration be used [2].

(1) A stock standard solution containing 50 mmol/L is prepared by dissolving 1.6561 g of SRM 928 in ion-free water. If the solution is cloudy or a precipitate forms, add a few drops of ammonium hydroxide. Mix, dilute to 100 mL in a calibrated volumetric flask and transfer immediately to a previously acid-washed, water-rinsed, dry polyethylene bottle. This solution is stable for six months [2].

The above directions are directly quoted from the reference given. Users are cautioned that carbonate-free ammonium hydroxide, not in excess of 0.2 ml., should be added. Otherwise, insoluble basic lead salts will form. However, experience at NIST indicates that this material will easily dissolve without cloudiness when high-purity water is used.

(2) An intermediate solution containing 500 μmol/L is prepared by a 1:100 dilution of the above stock solution. This solution may be stored in a capped polyethylene bottle at room temperature for one month [2].

(3) Working standard solutions of 0.5, 1.0, 2.5, and 5.0 μmol/L should be prepared each time an analysis is performed [2].

Note: Dilute aqueous lead standards remain stable for less than 3 h. Lead is readily adsorbed on the surfaces of glass and plastic containers and this reaction is accelerated by exposure to light, particularly ultraviolet light [3]. It is recommended that very dilute aqueous lead solutions be prepared in a darkened room and protected from light [4].

Source of Material[(1)]: The lead nitrate used for SRM 928 was obtained from the J.T. Baker Chemical Co. (Phillipsburg, NJ). This material was examined for compliance with the specifications for reagent grade lead nitrate as given in Reagent Chemicals, 5th edition, published by the American Chemical Society. The material met or exceeded the requirements in every respect.

REFERENCES

[1] Catanzaro, E.J.; Murphy, T.J.; Shields, W.R.; Garner, E.L.; *Absolute Isotopic Abundance Ratios of Common, Equal-atom, and Radiogenic Lead Isotopic Standards*; J. Res., NBS 72A, pp. 261–267 (1968).

[2] Kopito, L.; Shwachman, M.; *Measurement of Lead in Blood, Urine, and Hair by Atomic Absorption Spectroscopy*; In Standard Methods of Clinical Chemistry, Vol. 7; Cooper, G.R.; Academic Press Inc.: New York, NY, pp. 151–162 (1972).

[3] Kopito, L.; Shwachman, M.; *Determination of Lead in Urine by Atomic Absorption Spectroscopy using Coprecipitation with Bismuth*; Lab. Clin. Med., Vol. 70, pp. 326–332 (1967).

[4] Fundamentals of Clinical Chemistry; Tietz, N.W., Ed.; W.B. Saunders Co.: Philadelphia, PA, pp. 852–857 (1970).

Certificate Revision History: 23 February 2016 (Editorial changes); 10 February 2016 (Editorial changes); 05 April 1994 (Editorial changes); 27 February 1976 (Editorial changes); 01 November 1975 (Reprint); 15 June 1975 (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

[(1)] Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.